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VOL. 30. NO. 243. MARCH 1944.

PROPERTIES AND UTILIZATION OF PETROLEUM WAXES.

By C. G. GRAY.

THE waxy materials derived from petroleum are classified * into three main groups: (1) the paraffin-wax group; (2) the petrolatum group; and (3) the petroleum ceresin group. This classification is based mainly upon crystal size, consistency, and method of manufacture. Although both natural and artificial mixtures of the three classes are encountered, so that borderline cases exist, nevertheless the three classes possess distinctive properties, so that in general each class is particularly suited to certain specialized types of application. It will therefore be convenient to consider the properties and applications of each class separately.

1. THE PARAFFIN-WAX GROUP.

General Properties.

The waxes in this group are solids with a relatively pronounced crystalline structure. They consist mainly of mixtures of normal paraffin hydrocarbons in the range C_{18} to C_{35} approximately. Their setting points range up to about 160° F., and their mean molecular weights are normally in the range 225–450. In practice they are almost invariably derived from distillates.

Although no strict line of demarcation is laid down, a distinction is drawn between *refined paraffin wax* and *paraffin scale wax*. Refined paraffin wax is a paraffin wax of very low oil content, and is a colourless, translucent wax which is not more than slightly greasy to the touch. It is practically odourless and tasteless. Refined paraffin waxes may have setting points ranging from about 90° to 160° F. Scale wax, on the other hand, may contain a considerable proportion of oil, and its solid components normally cover a wider melting range than those of refined grades, which, in fact, are usually manufactured from scales by sweating, or fractional melting, to remove practically all the oil and to separate the solid components into cuts of narrower melting range. The setting point of scale wax may range from about 90° to 140° F. Scale waxes may be oily to the touch, and are frequently crumbly or flaky in texture.

Physical Properties.

Paraffin waxes melt to clear colourless liquids of low viscosity.

The density of the solid varies between the approximate limits of 0·90 and 0·94 at 20° C. On melting, there is considerable expansion, the density of molten paraffin waxes being about 0·77 just above the melting point.

The principal thermal properties are as follows: Specific heat of solid, about 0·5, but higher values may be shown at temperatures near the melt-

* Report of Nomenclature Panel of I.P. Committee No. 8 (Petroleum Wax), *J. Inst. Petrol.*, December 1943, **29**, (240), 361.

ing point, owing to the absorption of heat energy by crystalline changes. The specific heat of the liquid is given by the approximate expression $C = 0{\cdot}492 + 0{\cdot}0009t°$ C. at temperatures between the melting point and 300° C. The latent heat of fusion is about 40 cals./gm. The heat conductivity of the solid increases very rapidly with temperature, being about 0·00023 at 0° C. and 0·0006 at 25° C., in units of cals./sec./cm. cube/°C.

The electrical resistance of paraffin wax is very high—of the order 10^{17} ohms/cm. cube at room temperature. It shows a general rapid decrease as the temperature rises to the melting point, but once again crystalline transitions give rise to irregular variation as the melting point is approached. The dielectric constant is about 2·0.

Paraffin wax is completely insoluble in water, and is not readily wetted. It is to some extent soluble in petroleum fractions, turpentine, chlorinated solvents and, generally, in non-polar organic solvents. The solubility in, *e.g.*, white spirit increases rapidly with temperature. The wide variations possible in grades of wax and in composition of solvents makes it impossible to lay down definite limits, but a typical case is that of a 135–140° F. setting-point wax, which dissolves to the extent of 2 per cent. in an aromatic white spirit at 15° C., and to the extent of 46 per cent. weight at 40° C. D. S. Davis (*Industr. Engng. Chem., Ind. Ed.*, 1940, **32**, 1293) gives a nomograph relating the solubility of paraffin wax in petroleum fractions to the properties of the wax and of the solvent.

Chemical Properties.

Paraffin waxes are very stable chemically, a fact to which the name "paraffin" is due. They resist the action of concentrated acids and alkalis. At elevated temperatures they react with oxidizing or halogenating agents. The oxidation products are mainly fatty acids and hydroxy acids. Halogenation gives rise to substitution derivatives, those of higher halogen content being viscous liquids.

When burned under suitable conditions, paraffin wax gives rise to a clear flame, which is nearly white, smokeless, and odourless.

Applications of Paraffin Waxes and Scales.

In the years immediately prior to 1939, the world consumption of paraffin wax had reached the total of 500,000 tons per annum. Practically the whole of this quantity was absorbed in the manufacture of candles, waxed paper and cartons, polishes, matches, and electrical insulation materials, the distribution of the demand among these applications being approximately as follows:—

	Per cent.
Candles, nightlights and tapers	64
Waxed paper and cartons	15
Polish materials	10
Matches	5
Electrical insulation	3
Various other applications	3
	100

The demand for white scale wax is small in comparison with that for refined grades, most of the scale wax disposed of being used for the manufacture of nightlights and candles, and in minor applications such as barrel waxing.

(1) *Candles, Nightlights, and Tapers.*

Modern wax candles are manufactured by allowing the molten wax mixture to solidify in tubular tin moulds, the wicks being held centrally in the moulds while the wax is solidifying.

Since temperature control is of considerable importance during moulding, the moulds are surrounded by a water-bath, whose temperature may be adjusted by the introduction of cold water or of water which has previously been heated by injecting steam into the feed-line. The moulds are mounted vertically between metal end-plates which form the top and bottom of the water-bath. The bottom of each mould consists of a close-fitting piston, which is drilled centrally to allow the wick to be passed through it and upwards along the axis of the cylindrical mould. The upper surface of this piston is concave, and forms the tip of the candle, which is moulded in the inverted position. After solidification and cooling, the candles are ejected upwards from the moulds by raising the pistons. The candles are then gripped in a special type of clamp, and on lowering the pistons to the bottom of the now empty moulds, the machine is ready for moulding a new batch of candles. Fresh wick has been drawn from spools below the moulds, and is now supported centrally by the pistons at the lower end and by the finished candles at the upper end. The moulds are now refilled by the operator, the wax being allowed to flow from a pail over the top plate of the machine until all the moulds are filled, and a layer of wax about half an inch deep covers the plate, to allow for shrinkage into the moulds during solidification and cooling. The cooling water is usually maintained at 50–70° F., according to the wax being used. The wicks joining successive batches of candles are severed by a special knife when the new batch has solidified to an extent which makes support of the wicks from outside unnecessary.

The rack containing the finished candles is then removed and emptied. Before ejecting the next batch from the moulds, it is necessary to trim off the excess wax which covers the top plate of the machine and which still joins the bases of the candles. This operation is carried out by means of a broad spudding chisel when the wax is still plastic enough for easy handling, but cool enough to ensure that further shrinkage into the bases of the candles will be negligible. The excess portions of wick are removed with the wax fillet, which goes back for reboiling.

Fig. 1 shows in diagrammatic form the construction and operation of a single unit of the moulding machine. The modern machine contains up to 518 such moulds, together with water-bath, wick-bobbin compartment, hand-operated ram for raising the pistons, removable rack for holding the finished candles on ejection, and water and steam lines for temperature adjustment. Special types of moulds may be used to produce candles with fancy shapes, or with shaped butts.

Refined grades of paraffin wax with setting points of 120–145° F. are most commonly employed for candle manufacture. The setting point of the wax to be used must be chosen with due regard to the temperature con-

ditions which are likely to apply where the candle is to be stored and used, these conditions in turn depending on the geographical location of the market. Too low a setting point gives a candle which softens and may bend during storage or use; too high a setting point, on the other hand, gives a candle which is difficult to light and which burns with a very short wick and a small flame.

There is now a considerable amount of evidence showing that the composition of the wax also plays an important part in determining its suit-

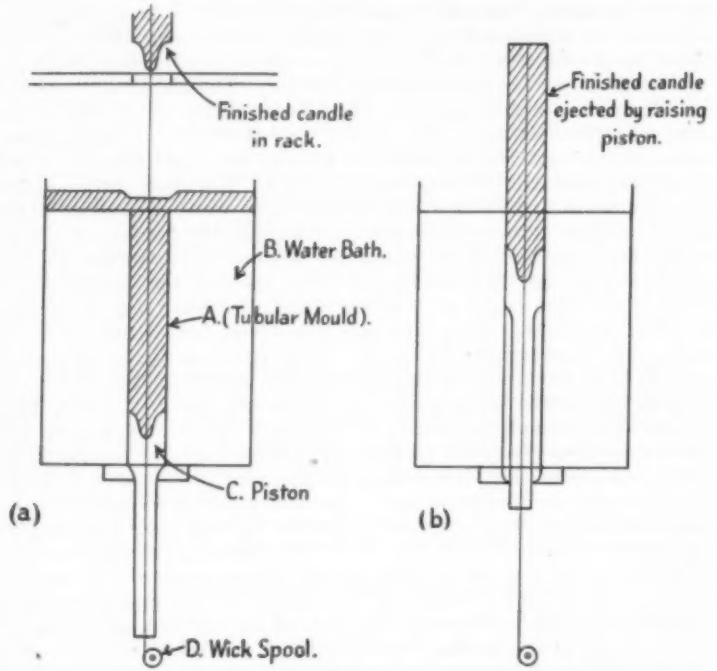

FIG. 1.

DIAGRAM OF SINGLE UNIT OF CANDLE-MOULDING MACHINE.

ability for candle manufacture. For example, the moulding characteristics, tendency towards cracking and mottling, and tendency to give plastic flow at room temperatures, all of which are properties which must be taken into account by the candle manufacturer, are bound up with the degree of fractionation of the wax, and not merely with its setting point.

It is now normal practice to add a proportion of stearic acid to the paraffin used for candle-making. The proportion of stearic acid may vary according to special requirements, but is usually of the order of 5 per cent. in the case of paraffin wax of low setting point (120° F.). The proportion may be reduced in the case of higher-setting wax. The stearic acid improves the appearance of the candle, giving the wax a smooth, marble-like

appearance. Moreover, it improves the strength of the wax, giving better "snap" at low temperatures and increased resistance to bending (plastic flow) at high room-temperatures. Contrary to the opinion which is sometimes expressed, the addition of stearic acid to paraffin wax in the proportions indicated usually produces a mixture whose melting point is lower than that of the original paraffin wax, so that the beneficial effects of the stearic acid on the mechanical properties of paraffin wax should not be confused with increase of melting point.

The wax and stearic acid are melted together by means of open steam, and since it is necessary to ensure that the moulding material is quite free from infusible solid particles of any kind, the melting and boiling are carried out over weak sulphuric acid, which destroys traces of metallic stearates and absorbs any fibrous particles which may have found their way into the wax or stearic acid from bags or packing materials. At this stage, too, the "reboilings" are added, containing portions of wick material, which are removed in the process of reboiling and settling. After melting and boiling, the mixture is allowed to settle, and when it has cleared it is run off into the steam-jacketed pans, from which the moulding-machine operator fills his pail.

The wicks are of cotton, and are plaited from three strands. The number of threads in each strand is varied in accordance with the size and type of candle being made. The nature of the wick influences the rate of consumption of wax, which in turn must be correlated with the diameter of the candle. Before use, the wicks are bleached and washed, and are then "pickled" in a solution of inorganic or organic salts, centrifuged and dried. The pickling salts commonly used include borax, ammonium phosphate, potassium nitrate, etc. Their purpose is to reduce char formation on the wick during burning and to eliminate smouldering of the wick on extinction of the candle. The pickling process may also be adjusted to influence the degree of curvature taken up by the wick while the candle is burning, this being of great importance in obtaining a candle which burns clearly and steadily with a good flame. The wick curvature is also affected by the weave of the wick and by the setting point of the wax employed.

Candles made by standard methods may vary in size from the tiny decorative "birthday cake" lights on the one hand, to church candles 60 inches high and 2½ inches in diameter on the other. References to larger ceremonial candles are encountered (for example, a candle 18 feet high and 7 feet in circumference is burnt in Naples for one day each year in memory of Caruso). Such candles, however, are presumably constructed by special methods, and it is doubtful whether paraffin wax enters into their composition to any great extent.

Nightlights are designed to burn more slowly than candles, giving a feebler illumination over an extended period. They may be moulded in a machine generally similar to the candle-moulding machine, except that the wick is not in place during moulding. Instead, a wire extends upwards through the mould, leaving a central hole in the nightlight, into which the wick is inserted by hand. The fine cotton wick is coated with a high-melting wax which stiffens it so that it can be inserted readily. This coating also supports a portion of the wick during burning and prevents its collapse into the cup of molten wax.

Two main varieties of nightlights are made, respectively with and without paper cases. In each type the wick is fastened to a small sheet-metal sustainer. In the manufacture of paper-cased lights, the wick and its sustainer are fastened to the prepared case by means of a hard wax of the sealing-wax type, the wax blank then being slipped into the case with the wick threaded through it. Uncased lights, which are designed to be burnt in glasses or in various appliances such as continental teapot warmers, are prepared simply by inserting the wick and its sustainer directly into the wax blank.

An alternative method for the manufacture of nightlights consists in warming the wax until it is plastic, and rolling or extruding the plastic mass into rods of approximately the required diameter, which are then cut into portions, each of which contains sufficient wax for one nightlight. These portions are then pressed to shape in steel moulds, under hydraulic pressure, giving blanks, which are finished off by inserting the wicks and fitting the nightlights into paper cartons.

Nightlights are made from paraffin wax alone, without the addition of stearic acid. A low-melting grade of wax (about 120° F.) is normally used, and white scale wax may be incorporated.

Wax tapers are made by passing the wick material through successive baths of molten wax, the wax coating being built up in stages and controlled by passage through a die of the appropriate size, after each dipping. The conditions are adjusted to ensure that the wax is in the plastic condition when it passes through the dies.

(2) *Waxed Paper and Cartons.*

Modern methods of production and marketing, whereby perishable goods manufactured in one hemisphere may be destined for consumption in the other, have brought with them the need for new and greatly improved wrapping materials, impervious to air, water, and water vapour, odourless, tasteless, physiologically inactive, robust enough and cheap enough to be commercially practicable. Waxed paper and cartons have obvious limitations as regards robustness, but fulfil all the other requirements. For certain applications in which great strength and rigidity are not essential, therefore, their use is extending very rapidly.

Waxed paper alone may be used as a wrapping material for a great variety of products such as bread, confectionery, beef-extract cubes, soap tablets, and safety-razor blades. Again, many proprietary materials are packed in unwaxed or lightly waxed cartons or boxes, which are then wrapped and sealed in waxed paper. This method is customary in the case of dry, granular products such as breakfast cereals, macaroni, salt, etc. It is also commonly used for biscuits. Lastly, liquid or semi-liquid products are conveniently handled in heavily waxed sealed cartons without any additional wrapping. The last method is familiar in the case of milk, honey, syrup, sandwich pastes, and so on.

Three main types of waxed paper may be distinguished: (1) impregnated or "dry waxed" paper in which the wax is completely absorbed into the paper web; (2) surface waxed on one side only; (3) surface waxed on both sides ("self-sealing paper"). The first type is used almost exclusively for retail wrapping of moist or greasy foodstuffs, and is therefore

widely known as Delicatessen Paper. Paper waxed on one side is occasionally used for wholesale packaging; in general, however, double-waxed self-sealing paper finds the widest application. Possibly the largest single application of this type of paper is for wrapping of bread. In certain countries, notably the United States of America, wrapped bread is the rule rather than the exception. When using this type of paper, the package is hermetically sealed by folding the wrapper so that the edges overlap, then hot-pressing the overlap so that the wax melts momentarily and seals the flap on resolidification. The operation is carried out on specially designed machines with continuous conveyor feeds.

Paper-waxing machinery varies greatly in design and method of operation, but two basic types may be distinguished, according to whether the wax is applied to the paper by waxed rollers or by total immersion of the paper in molten wax.

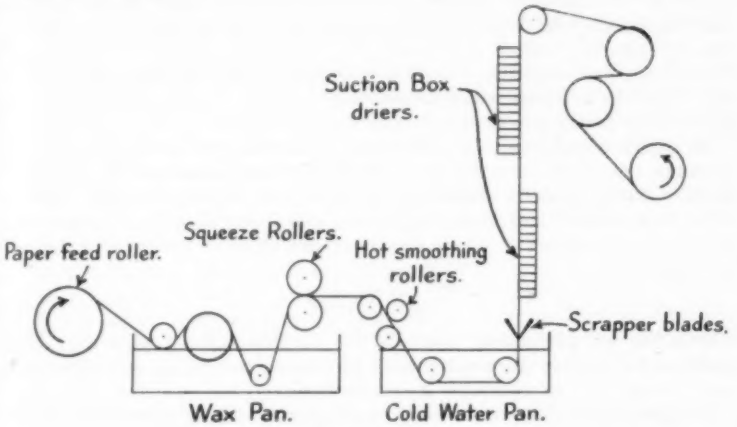

FIG. 2.

DIAGRAM OF PAPER-WAXING MACHINE.

Needless to say, single-waxed paper is produced by roller waxing. By suitable arrangements for turning the paper strip as it is fed through the machine, double waxing may be carried out in two stages on this type of machine. There are also machines in operation in which the paper is waxed on both sides simultaneously by passage between rollers, the lower roller rotating half-immersed in the wax-bath, and the upper one being fed with molten wax by means of a pump.

In the immersion type of waxing machine (see Fig. 2) the paper strip is fed from the roll over heated rollers and then through the wax-bath. On leaving the wax-bath, the paper passes through adjustable "squeeze rollers," which control the amount of wax to be left on the paper. It then passes through smoothing rollers and into a chiller, where the wax film is rapidly solidified. The speed at which the machine operates is extremely important. Surface-waxing machinery operates at high speeds, so that squeezing and chilling take place before the molten wax has been absorbed

into the paper web. A different procedure is adopted when "dry waxed" paper is being produced. Here the wax is retained in molten form by passage of the strip between hot rollers, so that absorption into the paper may take place.

The chilling apparatus commonly employed also falls into two main types, using cold-water immersion in one case and passage over chilled rollers in the other. Where water chilling is used, it is necessary to remove any water which may adhere to the surface on leaving the bath. This may be accomplished by air-suction gates, as shown in Fig. 2, or by passing the paper in light contact over polished metal doctor blades.

In general, rapid chilling produces opaque coatings with high gloss. Impregnation produces translucent papers without pronounced gloss. The finished paper may contain up to about 40 per cent. of its total weight in wax.

Cartons for liquids are made up into their finished shape before waxing. The waxing may be done by total immersion in molten wax, or by simultaneous spraying outside and inside, or, if desired, on the inside only, as the cartons pass the wax jets on a conveyor. After waxing, the cartons are chilled in a refrigerating compartment, or by dipping in cold water, followed by draining and drying.

As a general rule, only refined grades of paraffin wax, with very low oil contents, are satisfactory for paper waxing. The presence of oil, or inadequate refining, leads to deficiencies such as low sealing strength, flaking, poor gloss, colour- and odour-instability, and "blocking" (*i.e.*, tendency of the waxed surfaces to adhere and flow together when the paper is stacked or rolled).

(3) *Polishes.*

Paraffin wax is widely used in the manufacture of polishes. Wax polishes for various purposes consist of pastes, creams, or emulsions of a wax composition in a suitable liquid medium.

A paste of paraffin wax in white spirit or turpentine prepared by cooling a hot solution of the wax is not satisfactory as a polishing material. The lustre obtainable is not high, the film is soft and smears easily, the solvent is only loosely held and squeezes out readily, and the paste itself dries out too rapidly. Best results from all points of view are obtained by using blends of paraffin wax with other waxes, notably carnauba, candelilla, beeswax, and ozokerite or petroleum ceresin. In the finished compositions, the function of the hard waxes is to confer lustre and non-smearing properties to the surface; that of the paraffin wax is to plasticize the hard waxes, thus giving the surface good rubbing properties. The paraffin wax also plays its part in giving suitable "body" and structure to the paste, so that evaporation of solvent does not take place too rapidly in the tin, nor too slowly on the surface to be polished.

In the manufacture of paste polishes the waxes are melted together and run into a mixer, where they are blended with the solvent, which is normally white spirit, with or without a proportion of turpentine, to which the appropriate dye or colour is added. The mixture is fed to the filling machine after its temperature has been suitably adjusted to give the required consistency. The tins or containers are passed to the machine on trays

or on a conveyor belt. Alternatively, the machine may be run on rails over the laid-out tins. Delivery into the tins may be controlled automatically or by hand. The filled tins are cooled in air, or by passage through a cooling compartment, after which they are lidded and packed.

Liquid or solid creams are made by emulsifying a wax-solvent mixture with water. The emulsion may be of water-in-oil type or of oil-in-water type, according to the emulsifier used and the proportions of the phases.

A further type, widely used in America, popularly known as "dry bright," consists of a purely aqueous colloidal solution of wax and shellac.

(4) *Other Applications of Paraffin Wax and Scales.*

In the manufacture of matches, a low-melting paraffin wax (about 105–108° F.) is used to impregnate the matchsticks to assist their kindling and burning.

Various electrical appliances and equipment, such as condensers and cables, may incorporate paraffin wax or waxed paper as dielectric or insulator.

A number of chemical manufacturing processes make use of paraffin wax as a starting material. On oxidation it yields fatty acids, from which it is possible to prepare alcohols and synthetic fats. Chlorinated paraffin waxes find a number of applications—for example, as plasticizers for synthetic rubbers, as fireproofing agents, etc.

The impermeability of wax films to air, moisture, and bacteria underlies the use of wax for the preservative coating of fruit, vegetables, eggs, cheeses, hams, and other perishable goods.

In the Eastern world considerable quantities of paraffin wax were used in making brightly-coloured Batik cloths. The cloths are dyed in successive baths of colour with appropriate parts of the pattern blanked off at each stage by impregnation with wax. The wax is removed in boiling water between each dyeing stage, and the cloth then re-waxed with a new section of the pattern exposed for the next colour.

In the textile world, paraffin-wax rings are used to lubricate threads before spinning. Wax emulsions are also applied to fabrics to assist calendering after they are laundered. Paraffin-wax solutions are used in the production of waterproof and showerproof fabrics.

In the large-scale production of poultry for table use, the birds are plucked and stubbed by dipping in molten paraffin wax, then stripping the wax off whilst it is still warm and plastic. The feathers and stubs are removed with the wax.

As a fusible structural material, paraffin wax finds application in mounting and strengthening museum and art objects, and in the manufacture of crayons, coloured pencils, plaster statuettes, and modelling compositions.

Minor surgical applications include its use in plastic surgery for filling cavities and reshaping deformities and injuries—*e.g.*, to the nasal bones. Molten wax is also used as a medium for applying heat treatment for rheumatism and other ailments.

Scale wax is used in a number of minor applications, such as leather dressing, manufacture of fire-lighters, wood impregnation, barrel waxing, and so on.

2. The Petrolatum Group.

General Properties.

The waxy materials included in the petrolatum group are derived mainly from residues. Their congealing points range from about 100° to 180° F., and in consistency they vary from salve-like to firm and plastic. They exhibit crystalline structure, but the crystal size is nearly always less than that of paraffin waxes. Petrolatums are subdivided, on the basis of oil content, into the two classes : (1) *Petrolatum Wax*, which is substantially oil-free petrolatum, and is a plastic solid possessing some degree of tackiness. The congealing point is generally below 190° F., and is often indistinct; (2) *Petroleum Jelly* (Petrolatum), which is a soft, salve-like petrolatum consisting of petrolatum wax together with a substantial proportion of oil.

Petrolatum Wax

The plastic consistency of petrolatum wax, combined with its inert nature, makes it suitable for a number of special applications, such as the impregnation of insulating tapes and cable-core wrappings. It is also used to some extent for the preservative coating of cheeses, hams, and other perishable foodstuffs. Some of the firm, high-melting grades have found useful applications in paper-coating and in polish manufacture, where their plasticizing properties are useful in giving flexibility to the harder waxes and, in the case of polishes, in assisting the plastic flow of the film which gives rise to good " rubbing " properties.

The adhesion of petrolatum wax to metal and other surfaces is good, and this type of wax is sometimes used for coating the interiors of metal cans and drums for liquids. Drums for rubber latex and the metal retail " beer cans " used in the United States are frequently treated in this way.

Petroleum Jelly.

Petroleum jelly finds application as a special lubricant, both alone and as a component of greases. Considerable quantities are used for impregnating the hemp cores of wire ropes and hawsers. It is used as a component of some explosive compositions and as a plasticizer for bituminous roofing materials. Many anti-rusting and anti-corrosion compositions contain petroleum jelly. Modelling compositions of the Plasticine type may also incorporate petroleum jelly, whilst the highly refined material is widely used as a base for a number of pharmaceutical and toilet preparations.

3. The Petroleum Ceresin Group.

General Properties.

Petroleum ceresins are hard, brittle waxes, usually derived from residues, and essentially free from oil. The congealing point is usually above 160° F. (71° C.), and may be as high as 205° F. (95° C.). Petroleum ceresins are crystalline, but the characteristic crystal size is very much smaller than that of paraffin wax. The colour may range from dark brown to white, with yellow and orange perhaps most common.

A notable characteristic of petroleum ceresins (as also of ozokerite

ceresin) is that a small proportion (0·5 per cent.) of the ceresin in paraffin wax may have the effect of greatly reducing the crystal size of the paraffin wax, with an attendant improvement in impermeability and in mechanical properties. Hot solutions of paraffin waxes which have been "doped" in this way may form smooth, creamy pastes on cooling, instead of crystalline aggregates.

Applications.

In general, the applications of petroleum ceresins are based upon its inertness, high congealing point, micro-crystalline structure, and its power of conferring this micro-crystalline structure on paraffin wax. Thus waxes for electrical insulating and dielectric purposes frequently incorporate petroleum ceresin. It is also used in the formulation of polishes and of pharmaceutical and other pastes and creams.

SOME IDEAS ABOUT CORRELATION OF TEST-ENGINE RESULTS.

By T. P. Sands, C. J. Livingstone, and W. A. Gruse *

The idea of correlation implies the need for a simple apparatus to find out what a more complicated one will do, and that introduces the concept of the prototype unit. In the field of engines, the procedure could be used to try out mechanical design, fuel, or oil. When mechanical construction is under scrutiny, a test will be simpler only if it is made simpler, and it can easily be more complicated. If all the mechanical parts of an engine are to be tried out together, then the best procedure is to run the whole engine; if, however, one feature is to be examined and changed at a time—say valve action as affected by cam shape and position—that portion of the working system can be isolated and studied as a unit. All the conditions which are important can be controlled and an answer written down. As a matter of fact, it would probably not be done that way, but in principle, at least, it *could* be done. If fuel is to be examined in some aspect or other, we can make simplifying assumptions, and examine the one feature of interest, whatever it may be. Actually, as soon as engines were developed far enough to be critical as to fuel, the engineers found it very much simpler to use a small and rather elementary engine in which the fuel burning could be surrounded by a controlled environment. We made our first modified Delco knock-testing engine in 1923, and the progress of twenty years has brought to a good state of development the C.F.R. gasoline and diesel engines. The problems of measuring fuel-rating numbers have been simplified and standardized very successfully. But about ten years ago we found that the problem had been over-simplified and that there were too many kinds of service which did not correlate with one kind of octane number. So now we have Research and Motor, 1C and 3C, rich and lean, and perhaps soon we may have other kinds of fuel ratings. This history of fuel-testing units and scales supplies a good example of how correlation between full-scale and small units can go astray.

In testing lubricating oil, the justification for simplified apparatus rises from the very large number of different engine types in use, from the implicit recognition that some common factors in all of them are influencing the way the oil fails, and from the further recognition that small and uncontrolled variations in these fundamental factors will influence the conduct of an oil from one engine type to another.

It is an old story that oil in engines can be spoiled by contamination (dirt, water, blowby gases) and by chemical change. We can forget about contamination, although in special cases it, too, can be studied by prototype methods. We are concerned with chemical change, and that is straight up the alley of testing with simplified engines. The changes are confined

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pretty much to oxidation and thermal cracking. Since oxidized products crack more easily and cracked products oxidize more easily, the two are tied closely together. The important factors in chemical change are time, temperature, concentration, and contact, and if one is to do a good job of theorizing on the subject, each influence must be factored out. Temperature and time, for instance, are not just crankcase temperature and change period, but the weighted sum of the time–temperature functions for oil in the crankcase, at piston-under-crown, in the ring belt, and so on. By and large from one engine to another, the extremes of all the fundamental variables are of about the same order of magnitude—that is, the range from flame temperature to outside air temperature, the time oil spends in the various parts of an engine, and the degree of mixing of air, blowby gases, and oil are not too different. Therefore, we can reason about the typical case and not be far wrong, even though we know that oil does not go to pieces in a big stationary diesel over a long time, and may yet be ruined completely in a small automotive gasoline engine in 36 hours.

To mean anything, correlation must be discussed in terms of the tests and units people are trying to correlate with at present. As a result of a whole set of influences, including the efforts of individual engine-builders to lubricate their own output and the pressure of the war emergency in the direction of setting up specifications for universal-type oils, we have three widely used tests or sets of tests. These are :

A. The 36-*Hour Chevrolet Test.*—Thanks to a good deal of cooperative effort and some careful analysis, this test has been about as well standardized as could be expected for a multi-cylinder production engine. Crankcase oil is kept at 280° F., speed is equivalent to 60 m.p.h., and power output is set to correspond to road load. Considering the general design features of the Chevrolet engine, this test can be regarded as one involving some acceleration of normal oil deterioration.

B. General Motors Diesel 500-*Hour Test.*—This procedure involves operating a Series 71 G.M. diesel engine for 500 hours under full load at 2000 r.p.m. and without oil change. The temperatures of the intake air, fuel oil, crankcase oil, and jacket water are closely controlled. Samples of the crankcase oil are taken at intervals throughout the test and examined for deterioration. The engine is dismantled and the parts are examined for varnish and sludge deposit; copper-lead bearing shells are weighed to estimate corrosion.

C. Caterpillar Tests.—Three tests are ordinarily made :

1. *The* 480-*Hour Endurance Test.*—A single-cylinder Caterpillar test engine is operated for 480 hours at 1000 r.p.m. and a specified B.M.E.P. (75 lb.). The jacket temperature is 180° F. and the oil temperature 150° F. The oil is changed every 120 hours. The engine parts are inspected for stuck rings, scratching, or cutting at the piston top, and deposits on the piston skirt; wear on piston and liner are determined by measuring.

2. *Run-In and High-Load Tests.*—The same engine is provided with a special precombustion chamber which directs the fuel flame against the cylinder wall at three points. Power output

is determined at a definite fuel rate, and three runs, of 3½ hours each, are made; the oil and piston are changed and the engine flushed between runs. The piston and liner are inspected for scratching or wear.

3. 120-*Hour Test; Hot-Box Test.*—A four-cylinder Caterpillar engine, set in a hot room, is operated for 120 hours. The conditions include temperature of oil to bearings 212° F. and exit-water temperature 200°. The output is about 37 B.H.P. at 1400 r.p.m. Two connecting-rod bearings are babbitt and two are copper-lead. The latter are inspected for corrosion or scratching, and the liners and pistons are rated for deposits.

From the standpoint of wider usefulness, there is one feature of several of these tests which can be criticized. It is the pass-or-fail interpretation of results; and oil is either satisfactory or not; it either corrodes or does not corrode bearings in the Chevrolet test, corrosion being defined in exact terms. It either passes or does not pass the Caterpillar and 500-hour tests. The utmost in gradation on results is found in the borderline cases. This is no doubt the right thing to do for the war emergency, but it appears to fail when regarded as the basis for a rational correlation scheme.

The desire for correlation is based on the need for definite hooking up of an accelerated test of some kind with "average service," so that in the ideal procedure 5 hours of an accelerated test would produce, say, 50 hours of average service, 10 hours of test, 100 hours of average service, and so on. There are two difficulties about this. Pass-or-fail tests, such as we have to-day, must first be converted into graded results. An oil must make a grade of 5, 10, etc. The other difficulty is in setting the factor which will convert the results to average-service figures, and here we at once run into the obstacle that there is no such thing as average service by the ordinary definitions. Service must include all the engine troubles, bearing corrosion, ring sticking, sludge separation, and so on, and as soon as one of these becomes evident in a job, the service is no longer average, again by the ordinary definitions. Most of us, subconsciously at any rate, consider average service to be light passenger-car use, but this can hardly be defended, because that kind of running really does not involve failures of any kind. Coming closer home, average service may mean a combination of the most extreme conditions encountered in each of the several heavily loaded units on the market. In terms of the oil tests used to-day, it means just that. A sober second thought will tell us that average service, even for heavy-duty units, cannot mean the above, except for war operations. For the latter, oil must be made better than the average necessity. In the accepted logic of the emergency, an oil must meet all the service conditions of all the kinds of units, because the loss of one vehicle could, in theory at any rate, have a large influence on the course of a military action.

However logical this mode of thinking is for war, it may not be the wisest in adopting a permanent way of evaluating lubricants in peace-time. When war is done, the basic considerations of overall cost reassert themselves, and the ideal becomes the greatest good for the greatest number at the smallest total expenditure of goods and labour. According to this ideal, it may be best to undergo one failure of one eccentric freight transport

truck so that 999 others may operate at maximum efficiency for the longest time. This statement is based on the idea that a product which functions satisfactorily under the most severe conditions of all the current units, very probably will not, except by a happy accident, be the best oil for all, or even for more than one of them. When an oil is compounded to reach a definite value of detergency, oxidation stability, and freedom from depositing tendency, the chemist who makes it up has probably sacrificed a greater excellency in, say, wear resistance, in order to attain the detergency required, and so on. Left unrestricted to develop an oil of high detergency for a certain engine and within certain cost limits, the chemist could do much better if he did not have to remember that at the same time he must maintain a certain level of freedom from bearing corrosion because the oil is to be used in another type of engine suffering from that complaint. We know enough about oxidation inhibitors to suspect that an oil stabilized with certain types of inhibitors for the severe crankcase condition of one engine may be over-stabilized for the ring-belt conditions of another engine.

The present situation on engine testing began with the setting up by Caterpillar of a test for oils to be used in its own engines. This test served to bring out indications of several qualities, but laid particular emphasis on detergency. The object was the proving of oils which would meet Caterpillar's ideas of good service under the extreme demands on Caterpillar tractors. Following this, the General Motors laboratory defined the Chevrolet test, which, somewhat by contrast, emphasized the demand for marked oxidation stability. The oils which passed this test were intended for engines operating at very high crankcase temperature. From this it was a logical step to the General Motors 500-hour test, in which an oil was evaluated chiefly for oxidation stability, and to a smaller extent for detergency. While the Caterpillar test and its specialized successors were interpreted only for Caterpillar engines, the Chevrolet test is ordinarily considered to have a good deal of significance as indicating what will happen in General Motors diesels when the same oil is used, although complete reversals are sometimes encountered. These might be due to the presence or absence of lead, or to the differing temperature levels and conditions in the two tests.

As a matter of interpretation, and based on our temporary definition of average service, the Caterpillar 480-hour test can be regarded as not accelerated, the hot-box and scratch tests as accelerated, while the Chevrolet and General Motors tests are to some extent in the accelerated class.

At the outbreak of the war, many of the engine laboratories in the country were equipped for these three types of tests, and a fair degree of success had been attained in developing oils to meet the requirements of each separately. Two or three oils were available which met fairly well the requirements of all the tests. The war effected an immediate consolidation of requirements. The 2–104A specification was set up to define a class of lubricants which could meet all the conditions of the three outstanding tests; actually the approval was based on Caterpillar and General Motors diesel trials alone, the Chevrolet being included later to bring out the effects in gasoline engines. It was believed by those who took this step that an oil which would meet the requirements of this specification would lubricate satisfactorily all the automotive equipment of the armed forces under the

most exacting conditions they were likely to encounter. The experience to date has indicated that this judgment was correct. It must be recognized that the action was an emergency step, the best that could be done at the moment. But it was not based on theory or on an adequate knowledge of correlation with service. The fact that the armed forces have not experienced any particular trouble to date does not necessarily mean that the tests mentioned actually do correlate with military service demands. It means that we now have oils which are satisfactory under all the conditions, but we do not know how far the mark may have been overshot. Furthermore, we do not know whether, perhaps, certain types of power plants might be much better off from an operating standpoint with an oil which was not quite so good when used in other types of engines. An example of this, taken from industrial practice, is found in the choice of oils for the General Motors Series 71 diesel, and the Electromotive Model 567 as used in railway locomotives. These two appear to be structurally similar, except in size, but the speed, and consequently the temperature, are higher in the former. Originally a highly refined high V.I. oil was required for the Series 71 diesel, and it is still used, properly fortified by additives. For the Model 567 in the stream-lined trains, however, the operators have, in a high percentage of cases, selected a low V.I. uncompounded naphthenic oil as most satisfactory. The use in these latter units of an oil suitable for the Series 71 engines may or may not give satisfactory operation, but would appear to add an unnecessary load from the standpoints of operator economy and national resource economy.

At the moment we seem to have an opportunity to re-evaluate the situation to see whether, in some cases, scarce and valuable additives are being used in greater amounts than are really necessary. Lacking good data on correlation of our present three test types with actual military duty, and in the absence of information on actual work under combat conditions, it appears that progress along this line must be made slowly and with extreme care. All-round dependability is at the moment more important for military duty than efficiency and economy.

The most significant aspect of this situation from a long-time standpoint comes out in the lessons which we can draw for peace-time practice. In the first place, a definition of service is in order—the kind of service with which tests must correlate. We have said above that no one should be concerned about average passenger automobile duty—at least not until passenger-car engines are made much smaller and are habitually operated at high load factor. We have in mind rather the extreme demands on heavy-duty equipment. We repeat, that for military duty on one universal type of oil, average service must be defined as the sum of all the most severe conditions to be encountered in all the types of power plants employed; aircraft oils, for several reasons, cannot be included. Average heavy-duty peace-time service, on the contrary, is not a useful concept. One type of engine may be a bad actor from the standpoint of bearing corrosion, another develops a troublesome amount of varnish deposits, a third sticks too many rings, another has a bad record on oil economy, and so on. To average all these would define a total condition which could be met by a rather mediocre oil which would not measure up satisfactorily under the typical severe running conditions of any one of the units included in the average. At the

risk of being tiresome, we repeat that different units present different extreme requirements. The war-time answer is one oil good enough for all of them. But this almost certainly involves a great deal of sacrifice, justifiable only because it is a war-time answer.

The logical deduction from this—and it is an unpleasant one—is that a test must be set up for each accentuated engine trouble. That means a new engine test every time a new type of power plant comes on the market. Our laboratories would be more crowded than they are now with all sorts of test engines duplicating the conditions of every sort of obscure model.

Such an answer should not be necessary. It seems a not unreasonable hope that the development of a satisfactory prototype unit should be possible. This hypothetical engine, or its subtypes, should be flexible enough to permit operating under such conditions that a reproducible severe condition of varnishing tendency or of bearing corrosion or of wear could be set up. The general principles of that sort of manipulating are fairly well understood. The mode of operation would then be to rate oils in this engine as to detergency, oxidation stability, or wear on numerical scales; an oil would be rated 5 in detergency, 10 in oxidation stability, and so on. The engines which come on the market would be stated to have a need for 15 in oxidation stability, 5 in detergency, and so on. The analogy can be drawn with the present C.F.R. octane-rating engines; for the simpler cases we know that the octane requirements of different passenger-car engines can be stated in reasonably definite units. Of course, there are complications. Oxidation inhibitors, for instance, vary in efficiency, depending on test temperature, and detergents might do the same. An intelligent setting up of test conditions would be necessary, but the difficulty does not seem fundamental. This appears to be a rational and intelligent way out of the jungle of multiplication of test engines and one within the proper functioning of technical societies. At this stage of the situation it should be realized that tests must be developed primarily to correlate with service; exact duplication is less important than proper interpretation of results.

In passing, it seems desirable to offer one comment on the several engine tests mentioned in this paper. Within certain limits, and with very definite reservations, the Caterpillar single-cylinder engine seems to fall at least somewhere near the class of prototype units just proposed. It can remain the same, and need not be re-designed under stress of competition. Obviously, the test method will change from time to time, as field conditions change. The Chevrolet, however, a commercial power plant, is changed from year to year; several laboratories have had some little trouble in correlating results between Chevrolet models of different years. This makes it difficult to check current results with those of previous years, and it seems to us a very good reason why laboratory testing in commercial engines should not be promulgated by technical agencies. Testing in commercial engines has a definite field of usefulness; it includes the necessary transition from research to field operation, and it is definitely necessary in approval work by engine manufacturers in their own laboratories and with their own equipment. But for research and the correlation purposes discussed above, a standardized prototype seems more desirable, just as the C.F.R. is more desirable for octane numbers than a commercial Plymouth or Cadillac engine.

One final thought seems worth while. Engine tests should be divisible between those which are acceptance tests and those which are accelerated for information. It seems to us that acceptance tests should not be accelerated. The Caterpillar 480-hours is an acceptance test, it does not seem to involve more severe conditions than the oil will encounter in actual duty, and it appears to give quite reliable results—in fact, the final answer at 480 hours can often be predicted at 120 hours. The Caterpillar scratch test, however, is an accelerated one, and it does not always give the same consistent and reproducible answer. The point seems to us to be that when a test is accelerated, something more may happen than just acceleration. In theory acceleration simply speeds up what would happen if we did not accelerate. Actually, however, the ordinary ways of accelerating, such as raising the temperature, may start an entirely new tendency which did not exist at all at the lower temperature, and would never happen no matter how long the less severe condition was imposed. A simple example would be the application of an oxidation test to an oil containing a volatile additive. If the additive boils at, say, 250° F. and the normal testing temperature is 235°, then a test at 225° should be significant. But an attempt at acceleration by running the test at 275° would give an entirely misleading result. In the present state of our knowledge it seems wise to make acceptance tests under non-accelerated conditions and to reserve acceleration for information-seeking purposes only.

MR. L. RAYMOND writes: "I am entirely in sympathy with the writers' plea for prototype engines to simplify and rationalize lubricant and engine testing with the different types and makes of engines. It appears to me, however, that of greater importance than the development of prototype engines is the impartial collection and analysis of performance data to serve as the basis for establishing engine-test conditions to ensure correlation of laboratory results with field performance. Without these necessary basic data, the prototype engine will lose much of its value in predicting oil behaviour. The Caterpillar Tractor Co., in its development of the Class 1 Test particularly, has done an excellent job of facilitating laboratory work and its interpretation. The strength of this test lies not so much in the design and production of the prototype engine as in the specification of test conditions which correlate with the bulk of field performance in multi-cylinder Caterpillar engines. It is not my intention to minimize the value of prototype engines, but the availability of the engine will not by itself produce the desired effect. This is illustrated by the experience with the CUE assembly, in which a standard Wright Cyclone cylinder was used originally. Difficulty in obtaining oils of known service performance as a basis for correlation greatly retarded the development of the CUE assembly as a test tool for lubricant evaluation and engine study.

On page 73 of the paper, the authors mention the operation of the prototype engine under severe conditions of varnishing, bearing corrosion, or wear, with the oils being rated numerically in detergency, oxidation stability, etc., on the basis of these tests. I question whether arbitrary ratings of oils without complete definition of the test conditions will have the desired utility. This was emphasized forcefully in our laboratory by a rather extended study of bearing-corrosion effects in a single-cylinder engine operated under a variety of test conditions with some key oils. Changes in test conditions resulted in reversals in the order of merit of the oils in this one engine. Other data obtained in other engines showed wide variability in the corrosion tendencies of oils. Some of these data have been presented and reviewed by me in a paper published in the December, 1942, *S.A.E. Journal*. Similar variations in engine results arising from differences in time of test, use of leaded or unleaded gasoline, etc., have been observed and commented upon by others.

With respect to severity of test, I strongly agree that acceptance tests should not be accelerated to the point where significance of results becomes a matter of serious

concern. Tests such as the 36-Hour Chevrolet Test have the general weakness that they appear to be accelerated too much to be used with reliability in predicting or indicating the performance obtained in longer periods of operation under less severe operating conditions. Tests which are unsound, even though reproducible, have little merit. The major emphasis in the initiation of laboratory tests should be on significance of data, with subsequent attention to refinements in technique to improve precision. Too often the reverse method of attack has been the case.

I believe that the sound suggestion of the authors for the prototype engine deserves broad general support, but the inseparability of prototype engines and the basic field-performance data required for correlation must be recognized if effective utilization is to be made."

OBITUARY.

MR. J. H. C. DE BREY.

It is with deep regret that we have to announce the death, in Holland, of Mr. J. H. C. de Brey, on the 7th of October last, at the age of 63.

He was a man of outstanding ability and character. Not only did he contribute to the fundamental development of technical problems of the oil industry, but he also built up within the Royal Dutch Shell Group, from humble beginnings, a department through which he put his mark on the design of installations and plants throughout the world. Moreover, it was not only within the limits of the Shell organization that he was so well known, and there will surely be very many outside the Company who will have known him and appreciated his work.

It is not possible within the confines of this short notice to give a detailed description of his work, nor of the very wide scope of his accomplishments, but after a short outline of his life, we should like to mention some of the outstanding events in a rich and varied career.

De Brey was born in Rotterdam on the 18th November, 1880, and went to the elementary and secondary schools in that town. He then proceeded to Delft, where he obtained his degree as mechanical engineer in 1901 and electrical engineer in 1902.

From 1902 to 1906 he worked in the Caucasus on behalf of Messrs. Schuckert (as it was then) on the erection of electric power plants.

From 1906 to 1908 he worked in the Engineering Section of the Department of Public Works in Holland.

In 1908 he entered the service of the Royal Dutch Shell Group, and that same year went out to the Netherlands East Indies, where he acted as advisory engineer to the head office in Batavia. In this position he spent a great deal of his time on the oil-fields and in the refineries, and there acquired the basis of his experience for his later work.

In 1913 he returned to Europe, and after studying at first hand the oil-fields in Roumania, he went to Russia, where he was appointed Manager of the Group Company at Grozny.

In 1915 he returned to The Hague as Technical Advisor to the Board of Directors, and in the ensuing years built up the large organization which became known as the Technical Department, of which he became the head.

Whilst he spent the rest of his working days in this position, he made many trips to various places throughout the world.

Mr. de Brey was a member of various sections of the Netherlands Standardization Committee and an Honorary Vice-President of the second World Petroleum Congress, Paris, 1937. He was the founder of the Petroleum Division of the Royal Netherlands Institute of Engineers, of which division he also was the first president. Also for many years he was a member of the Institute of Petroleum.

Under the age limit imposed within the Group, he retired at the end of 1940, at the age of 60.

Owing to the occupation of the Low Countries, news since then of de

Brey has been scanty, but it is known that he suffered severely up to the time of his death.

During the five years of his stay in the N.E.I., de Brey was responsible for much original development work in connection with the recovery of natural gasoline from petroleum gases. In those days such compressor plants as were installed were used for air-lift purposes only. De Brey soon became aware of the possibilities of the benzine which could be obtained from such plants when operated on gas instead of air, and also installed field absorption plants for this purpose. The product obtained was, however, very wild and this must have directed de Brey's attention to the importance of conserving light fractions and to the necessity of stabilizing natural gasoline.

Another noteworthy achievement of his during his sojourn in the East was the development of a series of formulæ for the calculation of friction losses in oil pipe-lines. These formulæ were based on a large number of personally conducted practical experiments, and have been used in the Shell Group for a great many years. It is worthy of note that when, many years later, a new series of formulæ was developed, which took into consideration all the experimental data then available in literature, the deviation from de Brey's formulæ was remarkably small, indicating the excellence of his original experiments and their mathematical interpretation.

One of the many problems that came his way in 1917 was the design of a large pipe-line for a crude of very high viscosity. As no precedent of a mathematical treatment of the problem was known at the time, de Brey developed a method for the calculation of heated oil-lines, embodying the relation between quantity pumped, pipe diameter, and heat convection of the soil which, together with the earlier-mentioned friction formulæ, enabled him to predict friction losses with complete success.

His contact in the N.E.I. with gas recovery led him to search for a method for freeing natural gasoline from its most volatile unstable fractions—in other words, to stabilize it.

De Brey took out a patent on a pressure rectification apparatus, the stabilizer. Although in the U.S.A. the patent was not upheld, it is certain that de Brey's work gave a strong impetus to the general acceptance of the principle of stabilization, without which no refinery or gas-plant could now be imagined. The first pressure stabilizer in the Shell Group—and possibly in the world—was taken into operation in the N.E.I. around 1922.

A further development in the treatment of gases was the deep cooling of compressed gases, by expander engines, introduced in 1925, and by ammonia cooling.

Another innovation was the introduction of silica gel as a drying agent for natural gas, in conjunction with the above-mentioned installations.

The calculation and application of stabilizers gave rise to a more intensive study of close fractionation in general, which was influential in introducing better fractionation throughout the various refining centres in the Shell Group.

The keen realization of the importance of conserving the products—once produced—led de Brey to the study of the storage of light hydro-

carbons, and the development of pressure storage, in order to reduce breathing losses of tanks, combined with gasometers and compressor plants for the recovery of tank gases.

Another field in which de Brey did pioneering work was that of electrical dehydration. His assistance was invited during 1923, in connection with a field producing large quantities of a very stubborn crude-oil emulsion of high salt content, which had withstood all attempts at electrical or other treatment.

By means of a non-sinusoidal alternating current with a high peak-voltage relative to the mean voltage, developed specially for the purpose, de Brey successfully solved the problem and made the continuance of the oil-field in question possible as a commercial proposition.

He extended his work, in so far that he succeeded in reducing the residual salt content of the crude in question by a process embodying re-emulsification with fresh water and re-separation by electrical treatment. This principle, which was applied for the first time in 1926, had several years later gained much prominence in various parts of the world.

De Brey always had a clear vision of the manifold problems which presented themselves in the petroleum industry, and even in fields outside his own he possessed a remarkable gift for penetrating to the root of the question. Allied to this, his keen awareness of the necessity for research and of the importance of science in connection with a young industry like the petroleum industry, has, among others, borne fruit in the establishing of a special physical section of his department and of the " Proefstation Delft," a laboratory set up for the study of the practical application of oil products in engines, burners, etc.

Through his enthusiasm and clear conception of the meaning of research he was able to attract the right men to assist him in his task, and one need only mention names like those of Prof. Dr. W. J. D. van Dijck, the late G. D. Boerlage, and J. J. Broeze, to indicate the type of collaborator who, under de Brey's inspiring guidance, became known far outside their own organizations.

Besides his considerable technical abilities, de Brey also possessed the gift of conveying his ideas to others, which, together with his excellent mastery of languages, made him a valuable contributor at meetings and discussions in this country as well as others.

His numerous friends and acquaintances will be sorry to hear of his death, which occurred so soon after he severed his active connection with the Shell Group at the end of 1940, and which was certainly hastened by the conditions imposed by the occupation of his country.

His closer collaborators, and all those who have worked under his direction, will most vividly remember his energetic mentality and his remarkable ability for inspiring enthusiasm for the scientific development of our industry, which, combined with his gift for infusing a spirit of collaboration and team-work, made working with de Brey an experience not easily forgotten.

A. E. D.
J. K.
H. H. R.
M. L. K.